Application Note

An MDL study using Automated Solid Phase Extraction of Semi-Volatile Organic Compounds (AB 8270 SIM) in Water

Introduction

EPA Method 8270 is used to determine the concentration of semi-volatile organic compounds in extracts prepared from solid waste matrices such as soils, air sampling media and water samples. Extractions from water samples can be particularly difficult and time consuming due to the problematic analytes that need to be extracted, concentrated and analyzed. As well as the large quantities of solvent the manual sample process requires. Laboratories are required to report analytes at lower reporting limits than previously required, often needing to employ SIM scan variations of the method.

Given the recovery variability of 8270 compounds and the time it takes to conduct manual extractions, complete automation of the sample prep process is complicated. The following application outlines the entire automation of a modified 8270 extraction, delivering a complete, turnkey MDL study demonstrating both precision and accuracy for mixed classes of low-level analytes.

Instrumentation and Consumables

- FMS, Inc. TurboTrace® SPE system
- FMS, Inc. SuperVap® Concentrator system
- FMS, Direct-to-Vial concentrator tubes
- Thermo Certified GC/MS auto sampler vials
- Waters 1 gram Oasis HLB® Cartridge
- Restek 2 gram Sodium sulfate Cartridge
- Thermo Trace GC w/DSQ MS and AS3000Autosampler

Reagents

- Fisher Optima* Methanol
- Fisher Optima* Methylene Chloride
- Fisher HPLC Water
- Fisher Conc. Sulfuric Acid
- 8270 Standards

Procedure:

Sample Prep

Five, one liter samples are measured out in glass sample collection bottles

Each sample is spiked with relevant Standards

The sample PH adjusted to <2 with 6 N HCl

10 mL Methanol added to each sample bottle

TurboTrace SPE system

1. HLB Cartridges are conditioned with 10 mL of Methanol
2. HLB Cartridges are conditioned with 10 mL of DI H_2O
3. Samples are loaded across the HLB Cartridges via vacuum (~75mL/min)
4. Sample bottles are auto rinsed with DI water and the rinse loaded onto the HLB
5. The cartridges are dried with Nitrogen for 2 minutes
6. The HLB cartridges are eluted with 10 mLs of methylene chloride
7. The HLB cartridges are eluted with 10 mLs of methylene chloride
8. Cartridges are purged with N_2
9. Total time: 61.5 minutes

SuperVap Concentrator system

1. Pre-heat temp: 45 ºC
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 ºC
4. Nitrogen Pressure: 10 PSI
5. End point: 1mL

FMS
Fluid Management Systems

Results

Table 1: MDL data compiled over seven replicates.

Compound	Amount Spiked	SPE #1	SPE #2	SPE #3	SPE #4	SPE #5	SPE #6	SPE #7	Mean	STD Dev	MDL
Anthracene	0.05	0.0467	0.0510	0.0510	0.0500	0.0499	0.0515	0.0500	0.050	0.0016	0.0051
bis(2-ethylhexyl)phthalate	2	1.7920	1.9400	2.4600	1.8760	2.1200	1.9180	1.9040	2.001	0.2252	0.7076
dibenzo[a,h]anthracene	0.05	0.0401	0.0407	0.0438	0.0355	0.0394	0.0404	0.0407	0.040	0.0025	0.0077
Chloropyrifos	0.125	0.1450	0.1500	0.1638	0.1363	0.1638	0.1488	0.1475	0.151	0.0100	0.0313
Pyrene	0.05	0.0575	0.0580	0.0580	0.0555	0.0565	0.0565	0.0585	0.057	0.0011	0.0034
Dichlorobenil	0.125	0.0964	0.0986	0.0998	0.0961	0.0960	0.0976	0.1013	0.098	0.0020	0.0063
Dimethylphthalate	2	1.0760	0.9840	0.9300	0.8640	0.9580	0.9760	1.0200	0.973	0.0670	0.2104
Di-n-butylphthalate	2	1.9360	1.8960	2.0200	1.9420	1.9500	1.9200	2.0000	1.952	0.0437	0.1373
benzo[a]anthracene	0.05	0.0449	0.0471	0.0515	0.0463	0.0479	0.0464	0.0447	0.047	0.0023	0.0073
Chrysene	0.05	0.0415	0.0428	0.0458	0.0419	0.0425	0.0434	0.0424	0.043	0.0014	0.0044
indeno[1,2,3-cd]pyrene	0.05	0.0386	0.0398	0.0420	0.0357	0.0396	0.0396	0.0395	0.039	0.0019	0.0059
Phenanthrene	0.05	0.0560	0.0545	0.0540	0.0500	0.0525	0.0550	0.0560	0.054	0.0021	0.0067
benzo[b]fluoranthene	0.05	0.0469	0.0489	0.0525	0.0446	0.0462	0.0446	0.0471	0.047	0.0028	0.0087
2-methlynaphthalene	0.05	0.0476	0.0465	0.0425	0.0465	0.0424	0.0461	0.0474	0.046	0.0022	0.0069
benzo[a]pyrene	0.05	0.0434	0.0459	0.0500	0.0422	0.0429	0.0432	0.0442	0.045	0.0027	0.0084
Acenaphthylene	0.05	0.0378	0.0359	0.0317	0.0353	0.0355	0.0379	0.0407	0.036	0.0028	0.0088
Malathion	0.125	0.1288	0.1500	0.1650	0.1425	0.1600	0.1500	0.1450	0.149	0.0119	0.0373
Di-n-octylphthalate	2	1.6420	1.8580	2.3200	1.7600	1.9840	1.8860	1.8360	1.898	0.2143	0.6736
Acenaphthene	0.05	0.0431	0.0376	0.0349	0.0369	0.0390	0.0405	0.0468	0.040	0.0040	0.0127
Fluorene	0.05	0.0410	0.0384	0.0339	0.0369	0.0375	0.0392	0.0415	0.038	0.0026	0.0081
benzo[k]fluoranthene	0.05	0.0458	0.0479	0.0570	0.0447	0.0475	0.0483	0.0458	0.048	0.0041	0.0130
Diazanon	0.05	0.0555	0.0530	0.0565	0.0575	0.0615	0.0560	0.0595	0.057	0.0028	0.0087
Fluoranthene	0.05	0.0595	0.0585	0.0585	0.0545	0.0585	0.0565	0.0590	0.058	0.0017	0.0055
Butylbenzylphthalate	2	1.8800	2.0800	2.4600	2.0200	2.0800	2.0600	1.9660	2.078	0.1832	0.5757
Diethylphthalate	0.05	1.8340	1.5380	1.4020	1.4920	1.5720	1.5860	1.8800	1.615	0.1767	0.5553
Naphthalene	0.05	0.0430	0.0424	0.0408	0.0411	0.0400	0.0416	0.0455	0.042	0.0018	0.0057
benzo[g,h,i]perylene	0.05	0.0363	0.0355	0.0387	0.0334	0.0375	0.0364	0.0372	0.036	0.0017	0.0053
Prometon	0.125	0.1375	0.1198	0.1133	0.0984	0.1288	0.1209	0.1350	0.122	0.0135	0.0425

Conclusions

The results in Table 1 show the recoveries of all seven MDL replicates extracted on the FMS TurboTrace SPE/ SuperVap Direct-to-Vial Concentration system. The results demonstrate the ability of the FMS Total Sample Prep approach to deliver precise, consistent recoveries at ultra low level concentrations and with extreme precision. The combination of high recoveries and low standard deviations deliver a low-level MDL that is easy to achieve.

The FMS TurboTrace SPE and SuperVap Direct-to-Vial system totally automated turnkey solution is an ideal choice for automating a wide range of compound classes. The system is capable of extracting 5-30 samples in one program, allowing the system to grow with your laboratories throughput. Laboratories now have the ability to rapidly extract and concentrate directly to a vial, entire analytical batches using a turnkey system at the click of a mouse.

TurboTrace System and SuperVap Concentrator

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